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Toru TATSUMI

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for: "Apparatus for Vapor Phase Growth of Metal Oxide Dielectric Material"

VERIFICATION OF TRANSLATION

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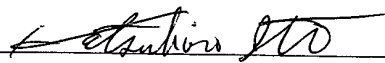
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[Inventor]
[Address or Domicile] c/o NEC Corporation, 7-1,
Shiba 5-chome, Minato-ku, Tokyo
[Name] Toru TATSUMI
[Applicant for Patent]
[Identification Number] 000004237
[Name or Appellation] NEC Corporation
[Agent]
[Identification Number] 100070219
[Patent Attorney]
[Name or Appellation] Tadashi WAKABAYASHI
[Telephone Number] 03-3585-1882
[Agent]
[Identification Number] 100100893
[Patent Attorney]
[Name or Appellation] Masaru WATANABE
[Agent]
[Identification Number] 100088328
[Patent Attorney]
[Name or Appellation] Nobuyuki KANEDA

[Agent]

[Identification Number] 100106138

[Patent Attorney]

[Name or Appellation] Masayuki ISHIBASHI

[Agent]

[Identification Number] 100106297

[Patent Attorney]

[Name or Appellation] Katsuhiko ITO

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[Title of the Invention] APPARATUS FOR VAPOR PHASE GROWTH
OF METAL OXIDE DIELECTRIC MATERIAL

[Claims]

5 [Claim 1] An apparatus to be employed for vapor
phase growth of a metal oxide dielectric material on a
substrate in a vacuum chamber using organometal gases and
an oxidizing gas as raw material gases by brining these
gases into contact with one another, comprising: an
10 apparatus part to be brought into contact with raw
material gases and to be heated to the temperature equal
to or higher than the decomposition temperature of the
raw material gases; at least the surface of the apparatus
part to which the raw material gases contact being made
15 of a metal oxide dielectric material (hereafter referred
to as a high temperature part-coating dielectric
material) same or not same as the metal oxide dielectric
material to be grown.

 [Claim 2] An apparatus for vapor phase growth
20 according to claim 1, wherein the high temperature part-
coating dielectric material is a material selected from
the group consisting of SrTiO_3 , BaTiO_3 , PbTiO_3 , $\text{Pb}(\text{Zr},$
 $\text{Ti})\text{O}_3$, $(\text{Ba}, \text{Sr})\text{TiO}_3$, $(\text{Pb}, \text{La})(\text{Zr}, \text{Ti})\text{O}_3$, $(\text{Pb}, \text{Nb})(\text{Zr},$
 $\text{Ti})\text{O}_3$, and $\text{SrBi}_2\text{Ta}_2\text{O}_9$.

25 [Claim 3] An apparatus for vapor phase growth
according to claim 1 or 2, wherein the apparatus part to
be brought into contact with raw material gases and

heated to the temperature equal to or higher than the decomposition temperature of the raw material gases comprises a susceptor for supporting a substrate.

[Claim 4] A vapor phase growth apparatus to be
5 employed for vapor phase growth of a metal oxide dielectric material on a substrate in a vacuum chamber using organometal gases and an oxidizing gas as raw material gases by bringing the gases into contact with one another on the substrate, comprising: an apparatus
10 part to be brought into contact with the raw material gases and to be heated to the temperature equal to or lower than the decomposition temperature of the raw material gases; at least the surface of the apparatus part to which the raw material gases contact being made
15 of aluminum oxide.

[Claim 5] A vapor phase growth apparatus according to claim 4, wherein the apparatus part to be to be brought into contact with the raw material gases and heated to the temperature equal to or lower than the
20 decomposition temperature of the raw material gases is the inner wall of a vacuum chamber to carry out vapor phase growth of a metal oxide dielectric material therein.

[Claim 6] A vapor phase growth apparatus according to claim 4, wherein the apparatus part to be to be brought into contact with the raw material gases and heated to the temperature equal to or lower than the
25 decomposition temperature of the raw material gases

comprises a liner installed in the inner wall of a vacuum chamber to carry out vapor phase growth of a metal oxide dielectric material therein.

[Claim 7] A vapor phase growth apparatus
5 according to any one of claims 1 to 6, wherein the vapor phase growth apparatus comprises wall heating means to heat the walls of the vacuum chamber and is to set at a temperature equal to or higher than that allowing the organometal gases to have sufficiently high vapor
10 pressure and at a temperature equal to or lower than the decomposition temperature of each organometal gas.

[Claim 8] A vapor phase growth apparatus according to any one of claims 1 to 7, wherein the vapor phase growth apparatus comprises respectively independent
15 pipes as pipes for introducing respective raw material gases into the vacuum chamber.

[Claim 9] A vapor phase growth apparatus according to any one of claims 1 to 8, wherein the vapor phase growth apparatus comprises separately the vacuum
20 chamber and a heater chamber equipped with a heater for heating a substrate, and the vacuum chamber and the heater chamber are independently provided with pumps for exhaust gases.

[Claim 10] A vapor phase growth apparatus
25 according to claim 9, wherein the vacuum chamber and the heater chamber are communicated with each other through holes for pins to exchange substrates mounted on a

susceptor and in the state the substrates are mounted on the susceptor, the holes are closed by the substrates to separate the vacuum chamber and the heater chamber.

[Claim 11] A vapor phase growth apparatus
5 according to any one of claims 1 to 10, further comprising: a main exhaust line connected to a pump and a sub exhaust line equipped with a water cooling trap between the vacuum chamber and a pump as exhaust lines for evacuate the vacuum chamber.

10 [Claim 12] A vapor phase growth apparatus according to any one of claims 1 to 11, wherein the vacuum chamber is made of aluminum.

[Claim 13] A vapor phase growth apparatus to be employed for vapor phase growth of a metal oxide
15 dielectric material on a substrate in a vacuum chamber using organometal gases and an oxidizing gas as raw material gases by bringing these gases into contact with one another on the substrate, comprising: a valve capable of opening at the time when a substrate is put in and
20 taken out between the forgoing vacuum chamber and an exchange chamber and with a movable shielding plate in the vacuum chamber side of the valve for preventing adhesion of raw material gases to the valve.

[Claim 14] A vapor phase growth apparatus
25 according to claim 13, wherein the movable shielding plate is set at a temperature equal to or higher than that allowing the organometal gases to have sufficiently

high vapor pressure and at a temperature equal to or lower than the decomposition temperature of each organometal gas.

[Claim 15] A vapor phase growth apparatus
5 according to any one of claims 1 to 14, wherein the vapor phase growth apparatus is used for formation of a capacitor film of a semiconductor device.

[Detailed Description of the Invention]

[0001]

10 [Filed of the Invention]

The present invention relates to a fabrication apparatus of a semiconductor device having capacitor elements, more specifically, the present invention relates to a vapor phase growth apparatus to be employed
15 in the case of carrying out vapor phase growth of high-dielectric-constant film and a ferroelectric film to be used for a capacitor and a gate of a semiconductor integrated circuit using raw materials of organometal gases.

20 [0002]

[Prior Art]

In recent years, ferroelectric memories using ferroelectric capacitors, dynamic random access memories (DRAMs) using high-dielectric-constant capacitors, and
25 the likes have actively been investigated and developed. These ferroelectric memories and DRAMs comprise selective transistors and store information using capacitors, as

memory cells, connected to one diffusion layer of each selective transistor. The ferroelectric capacitors are provided with, as a capacitive insulating film, a ferroelectric film such as $\text{Pb}(\text{Zr}, \text{Ti})\text{O}_3$ (hereafter referred to as PZT) or the like and enabled to store non-volatile information by polarizing the ferroelectric film. Meanwhile, the high-dielectric-constant capacitors are provided with, as a capacitive insulating film, a high-dielectric-constant thin film of $(\text{Ba}, \text{Sr})\text{TiO}_3$ (hereafter referred to as BST), so that the capacitors have high capacitance and enable elements to be miniaturized in a fine structure.

[0003]

In the case of using such ceramic materials for semiconductor elements, it is highly important to deposit such ceramic materials in a thin film with excellent crystallinity on a conductive film to be a lower electrode. As the method for depositing a thin film, there have conventionally been reported a sol-gel method, a sputtering method, and a CVD method.

[0004]

The sol-gel method is a method in which organometal materials dissolved in an organic solvent are applied to a wafer having a lower electrode formed thereon, by a spin coating, and crystallized by annealing in oxygen. In the method, since crystallization takes place in a solid phase, the temperature for the crystallization

leading to a ferroelectric film is necessary to be very high and in the case of using PZT for a metal oxide dielectric film, the crystallization temperature required to acquire sufficient ferroelectric properties is 600°C and in the case of BST, the crystallization temperature required to acquire sufficiently high dielectric properties is 650°C. A crystal formed by the foregoing method has a defect that the orientation of the crystal is uneven. Further, the sol-gel method is difficult to be applied to a wafer with a large diameter and furthermore, the method is inferior in coating of stepped surfaces and thus unsuitable for high integration of a device.

[0005]

The sputtering method is a method in which a film is formed on a wafer having electrodes thereon, by reactive sputtering of a ceramic sintered body to be formed into the film using Ar + O₂ plasma and then subjected to crystallization by annealing in oxygen. The film can be made even by making the diameter of the target wide and a sufficiently high film formation speed can be obtained by increasing the power for plasma generation. However, in the sputtering method as well, the crystallization is required to be carried out at a high temperature and in the case of using PZT for a metal oxide dielectric film, the crystallization temperature required to acquire sufficient ferroelectric properties

is 600°C and in the case of BST, the crystallization temperature required to acquire sufficiently high dielectric properties is 650°C. Further, in the sputtering method, since the composition of the film
5 obtained is substantially determined by the composition of the target used, the target has to be replaced in order to change the composition and that is disadvantageous from a viewpoint of production process.

[0006]

10 The CVD method is a method in which raw materials are transferred in gaseous state to a vacuum chamber to form a film. It is considered that the CVD method is excellent in uniform film formation on a wafer with a large diameter and in coating property on stepped
15 surfaces and highly expected as a technique for mass production in the case of application to ULSI. The metals of constituent elements of ceramics are Ba, Sr, Bi, Pb, Ti, Zr, Ta, La, and the likes and since proper hydrides and chlorides of these metals are very few,
20 organometals are used for vapor phase growth. However, these organometals have low vapor pressures and are mostly solids or liquid at a room temperature and, therefore, a transferring method using a carrier gas is employed.

25 [0007]

Fig. 9 is a diagrammatic illustration illustrating the reaction gas supply method in the case of using a

carrier gas. The apparatus is composed of a thermostatic tank 201, a bottle 202, organometal raw materials 203 made ready in the bottle, a supply pipe 204 for an inert carrier gas such as Ar, N₂, and the likes, a mass flow controller 210 for the carrier gas, a supply pipe 205 for organometal gases to be transferred by the carrier gas, a growth chamber 207 equipped with a heating mechanism 206, a gas discharge port 209, and so forth.

[0008]

10 The organometal materials 203 made ready in the bottle are, for example, strontium bis(dipivaloylmethanate) {abbr. Sr(DPM)₂}, barium bis(dipivaloylmethanate) {abbr. Ba(DPM)₂}, and lead bis(dipivaloylmethanate) {abbr. Pb(DPM)₂}, and maintain
15 solid or liquid state at a normal temperature. In the CVD method using the apparatus, these organometal materials are sublimated in the bottle 202 and supplied to the growth chamber 207 while being accompanied with a carrier gas supplied from the supply pipe 204 to form a
20 film on a heated wafer 208. The pressure during the film formation at that time is the atmospheric pressure or a pressure of several Torr.

[0009]

25 In the case such a method is employed, however, there is a disadvantageous point that the organometal gas flow rates in the carrier gas are difficult to be quantified and precisely controlled. That is because the

carrier gas contains organometal gases in an amount equal to or more than the amount corresponding to the saturated vapor pressure determined by the temperature of the thermostatic tank 202 and because the flow rates of the organometal gases depend not only on the flow rate of the carrier gas but also on the surface area of raw material solids and the temperature of the thermostatic tank. According to the description of Jpn. J. Appl. Phys. Vol. 32 (1993) P. 4175 on the formation of a film of PTO (lead titanate: PbTiO_3) by the above film formation method, the temperature of the formation of a PTO film is very high, as high as 570°C , and the film obtained has a drawback point that crystal orientation is uneven.

[0010]

In conventional production of a ferroelectric memory or a DRAM, the foregoing film formation methods are employed, however heating at a high temperature around 600°C or higher in oxygen atmosphere is essential and crystal orientation has been difficult to be controlled.

[0011]

To give a description of a semiconductor in terms of the structure, in order to make a ferroelectric capacitor and a high-dielectric-constant capacitor well functioning, it is required to electrically connect an electrode of either one of capacitors to a diffusion layer of a selective transistor. Conventionally, in a

DRAM, it is general that polysilicon connected to one diffusion layer of a selective transistor is used as one electrode and that a SiO_2 film, a Si_3N_4 film, or the like is formed as a capacitive insulating film on the surface of the polysilicon to compose the capacitor structure. However, since the ceramic thin film is an oxide, polysilicon is oxidized when the thin film is to be formed directly on the surface of the polysilicon and owing to that, it is impossible to form a good thin film. Hence, 1995 Symposium on VLSI Technology Digest of Technical Papers p. 123 describes a cell structure in which the upper electrode of a capacitor and a diffusion layer are connected by a local wiring of a metal such as Al or the like. Also, International Electron Devices Meeting Technical Digest, 1994 p. 843, describes a technique of forming a PZT capacitor on polysilicon using a TiN barrier metal. Regarding a DRAM, for example, International Electron Devices Meeting Technical Digest, 1994 p. 831, describes a technique of forming a STO (strontium titanate SrTiO_3) thin film on a RuO_2/TiN lower electrode form on a polysilicon plug to compose a capacitor.

[0012]

[Problem to be Solved by the Invention]

In order that an integration degree of an element employing a ferroelectric or high-dielectric material is comparable with conventional DRAM and flash memory, it is

necessary that a metal oxide dielectric film is formed directly on a plug provided to an interlayer insulation film or on a multiplayer metal wiring structure as a capacitor film. However, a satisfactory capacitor film
5 is hardly obtained by conventional vapor phase growth methods as described above. Furthermore, the problem of dust adsorption during the film formation makes the improvement in the integration degree a hard task.

[0013]

10 In any conventional film formation method, e.g. a sol-gel method, a sputtering method, and a CVD method, with the film growing, raw materials or products adhere to an apparatus inner wall part or to a susceptor part or the like holding a wafer. Subsequently, adhered raw
15 materials or products are parted and peeled off, then, these materials adhere to the wafer surface to cause a problem that device fabrication thereafter is obstructed.

[0014]

For example, in a sol-gel method, a raw material
20 solution is scattered to the apparatus inner walls at the time of spin coating and segments of a film of the dried scattered raw material adhere to the wafer surface. Also in a sputtering method, a material of a target adheres to the apparatus inner walls and being parted and scattered,
25 the material pollutes the wafer surface.

[0015]

In terms of a CVD method, in the case of a kind of

methods employing a furnace for heating a wafer put
therein by increasing the temperature of the walls,
organometal gases are decomposed in the heated walls and
the decomposed matter is scattered and pollutes the wafer
5 surface.

[0016]

The present invention, relating to a vapor phase
growth apparatus to be employed for the foregoing vapor
phase growth of a metal oxide dielectric film, aims at
10 providing a vapor phase growth apparatus capable of
forming a metal oxide dielectric film with little dust
adsorption by suppressing dust generation during the film
formation.

[0017]

15 [Means for Solving the Problem]

The present invention relates to an apparatus to be
employed for vapor phase growth of a metal oxide
dielectric material on a substrate in a vacuum chamber
using organometal gases and an oxidizing gas as raw
20 material gases by brining these gases into contact with
one another, comprising: an apparatus part to be brought
into contact with raw material gases and to be heated to
the temperature equal to or higher than the decomposition
temperature of the raw material gases; at least the
25 surface of the apparatus part to which the raw material
gases contact being made of a metal oxide dielectric
material (hereafter referred to as a high temperature

part-coating dielectric material) same or not same as the metal oxide dielectric material to be grown.

[0018]

Though the decomposed products of raw material
5 gases are deposited when the raw material gases are brought into contact with the part(s) which are heated at the temperature equal to or higher than the decomposition temperature of the raw material gases, parting of the deposited decomposed products does not take place in the
10 case where the parts to be brought into contact with the raw material gases are made of the high temperature part-coating metal oxide dielectric material and subsequently. Therefore, the generation of particles can be suppressed. It is also sufficient that, in the part(s), a portion of
15 surface thereof to be brought into contact with the raw material gases is coated with the high temperature part-coating dielectric material. Or, the whole body of the parts may be made of the high temperature part-coating dielectric material.

20 [0019]

The high temperature part-coating dielectric material may be the same or not the same as the metal oxide dielectric material to be grown. An apparatus of the present invention for vapor phase growth is suitable
25 for growing a metal oxide dielectric film on a substrate and since a ferroelectric material and a high-dielectric-constant material are preferable as the metal oxide

dielectric material to be grown, a ferroelectric material and a high-dielectric-constant material are also used as the high temperature part-coating dielectric material.

[0020]

5 The apparatus parts to be brought into contact with the raw material gases and to be heated to the temperature equal to or higher than the decomposition temperature of the raw materials gases include any parts which satisfy such condition and disposed in the
10 apparatus for the vapor phase growth. Among these, a susceptor for supporting a wafer is an example part representative of such apparatus parts. And at the same time, effect is the most prominent if the high temperature part-coating dielectric material is applied
15 to the susceptor.

[0021]

Further, in the present invention, the foregoing apparatus for vapor phase growth comprises wall heating means for heating the walls of a vacuum chamber and it is
20 preferable to set the temperature of the walls not lower than the temperature at which the organometal gases have sufficiently high vapor pressure and not higher than the decomposition temperature of the organometal gases.

[0022]

25 Further, the present invention relates to a vapor phase growth apparatus to be employed for vapor phase growth of a metal oxide dielectric material on a

substrate in a vacuum chamber using organometal gases and an oxidizing gas as raw material gases by bringing the gases into contact with one another on the substrate, comprising: an apparatus part to be brought into contact
5 with the raw material gases and to be heated to the temperature equal to or lower than the decomposition temperature of the raw material gases; at least the surface of the apparatus part to which the raw material gases contact being made of aluminum oxide. Since the
10 organometals of the raw materials hardly adhere to the faces made of aluminum oxide, particle generation can be suppressed.

[0023]

Such parts to be coated with or made of aluminum
15 oxide include any parts which satisfy such conditions as to be brought into contact with the foregoing raw material gases and to be heated to not higher than the decomposition temperature of the raw material gases. Among these, the inner wall is an example part
20 representative of such parts. And at the same time, effect is the most prominent if aluminum oxide is applied to the wall.

[0024]

Further, the present invention relates to a vapor
25 phase growth apparatus to be employed for vapor phase growth of a metal oxide dielectric material on a substrate in a vacuum chamber using organometal gases and

an oxidizing gas as raw material gases by bringing these gases into contact with one another on the substrate, comprising: a valve capable of opening at the time when a substrate is put in and taken out between the forgoing vacuum chamber and an exchange chamber and with a movable shielding plate in the vacuum chamber side of the valve for preventing adhesion of raw material gases to the valve.

[0025]

10 [Modes of Execution]

The apparatus of the present invention is effective to formation of a metal oxide dielectric film on a substrate. The metal oxide dielectric materials for a ferroelectric or high-dielectric-constant film formable to be a film by a film formation method of the present invention include oxides having general formulas;

15 $\text{SrBi}_2\text{Ez}_2\text{O}_9$ (wherein Ez denotes Nb or Ta); $\text{EpBi}_2\text{EqO}_9$ (wherein Ep denotes Ba or Pb and Eq denotes Nb or Ta); $\text{ExBi}_4\text{Ti}_4\text{O}_{15}$ (wherein Ex denotes Sr, Ba or Pb); $\text{Ey}_2\text{Bi}_4\text{Ti}_5\text{O}_{18}$ (wherein Ey denotes Sr, Ba or Pb); $\text{Pb}(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$; $(\text{Pb}_{1-y}\text{La}_y)(\text{Zr}_{1-x}\text{Ti}_x)\text{O}_3$; $\text{Bi}_4\text{Ti}_3\text{O}_{12}$; SrTiO_3 , and $(\text{Ba}_{1-x}\text{Sr}_x)\text{TiO}_3$.

[0026]

Preferred materials include SrTiO_3 , BaTiO_3 , $\text{Pb}(\text{Zr}, \text{Ti})\text{O}_3$, $(\text{Ba}, \text{Sr})\text{TiO}_3$, $(\text{Pb}, \text{La})(\text{Zr}, \text{Ti})\text{O}_3$, $(\text{Pb}, \text{Nb})(\text{Zr}, \text{Ti})\text{O}_3$ and $\text{SrBi}_2\text{Ta}_2\text{O}_9$.

[0027]

Thoses material are also suitable for use as the

high temperature part-coating material.

Practical examples are PbTiO_3 , SrTiO_3 , BaTiO_3 , $(\text{Pb}, \text{La})(\text{Zr}, \text{Ti})\text{O}_3$, $(\text{Pb}, \text{Nb})(\text{Zr}, \text{Ti})\text{O}_3$, $\text{SrBi}_2\text{TaO}_9$, and the likes.

5 [0028]

In the case a metal oxide dielectric material to be grown and a high temperature part-coating dielectric material are different, materials with the same crystal system (crystal structure) or similar crystal system to the system are preferably selected. For example, in the case of growing a perovskite type material, a perovskite type material is preferable to be used as the high temperature part-coating dielectric material. In the case of growing a metal oxide dielectric material with a layered structure such as $\text{SrBi}_2\text{Ta}_2\text{O}_9$, a material having a layered structure is preferable to be used as the high temperature part-coating dielectric material.

[0029]

The organometal material usable in the apparatus of the invention is not particularly restricted, so long as it exerts a desired vapor pressure when heated and provides a metal oxide dielectric material in a vapor phase by the reaction with an oxidizing gas. Examples include alkoxides such as i-propoxides and t-butoxides; dicarboxy methanates such as dipivaloylmethanate; and other conventional ones.

[0030]

The oxidizing gas gives a metal oxide dielectric material from organometal gases. For examples, the oxidizing gases include NO₂, ozone, oxygen radical, oxygen ion, and the likes.

5 [0031]

In the apparatus of the present invention, a metal oxide dielectric material is formed in a vacuum chamber using organometal gases and an oxidizing gas as raw material gases by bringing these gases into contact.

10 [0032]

The apparatus is so composed as to keep separated of the organometal gases and the oxidizing gas from each other before they are introduced into the vacuum chamber. The reason for that is, because if the organometal gases and the oxidizing gas are brought into contact with each other to result in generation of particles of metal oxides.

[0033]

A vapor phase growth apparatus of the present invention can be applicable to any purpose as long as metal oxide dielectric materials such as those described above are to be formed on a substrate. The substrate is in general a semiconductor substrate and includes a semiconductor substrate having element thereon. An apparatus of the present invention can be applicable for formation of, for example, capacitor films such as ferroelectric capacitor film and high-dielectric-constant

capacitor films or memories and the likes in which a ferroelectric film and a double layer structure of a silicon oxide film and a ferroelectric film known as MFS (metal ferroelectrics silicon) and MOFSMFS (metal oxide ferroelectrics silicon), and the likes.

[0034]

Especially, it is most preferable to employ the apparatus for the formation of capacitor films and the capacitor electrode to carry out vapor phase growth of a metal oxide dielectric film thereon is not specifically limited but, for example, the materials usable for the electrode are Pt, Ir, Ru, IrO₂, RuO₂, TiN, WN and the likes.

[0035]

Hereafter, an embodiment of the present invention will be described with reference to Figs.

[0036]

A schematic diagram of one example of a thin film vapor phase growth apparatus to be employed for the present invention is illustrated in Fig. 1. The present apparatus is composed of an exchange chamber 101, a vacuum chamber 102, and a raw material supply system 103 and a plurality of sheets of 8-inch wafers can be housed in the exchange chamber.

[0037]

Double gate valves 104 are installed between the vacuum chamber and the exchange chamber and the space

between the two gate valves is evacuated by a pump. A wafer transferring mechanism 105 is installed for transferring wafers between the vacuum chamber and the exchange chamber. With such a constitution, wafers can
5 be replaced and transferred without the vacuum chamber being exposed to atmospheric air. The exchange chamber is evacuated to 10^{-7} Torr by turbopumps 106 independently installed from the pump for the vacuum chamber.

[0038]

10 A schematic diagram showing a part of the cross-section of the vacuum chamber is illustrated in Fig. 2. The vacuum chamber 406 is equipped with a heater denoted with 416 and made of aluminum to be heated to a prescribed temperature. The material of the vacuum
15 chamber 406 is preferable to have a high thermal conductivity and, for example, aluminum and a stainless steel may be employed. Especially, aluminum, which has a high thermal conductivity, is preferable and by using aluminum, the inner walls of the vacuum chamber can
20 evenly be heated.

[0039]

Wafers 417 are so set on a susceptor 403 made of quartz as to keep the faces to be subjected to device formation upward. Three holes 401 with 5 mm diameter are
25 formed in the susceptor 403 made of quartz and pins 402 made of quartz are moved up and down through the holes to mount wafers on the susceptor 403 from the transferring

apparatus. After the wafers are mounted on the susceptor made of quartz, the holes for the pins are closed by the wafers themselves. The susceptor is so put on projections projected from a wall of a vacuum chamber
5 made of aluminum as to overlap the rim parts on the projections and in the state in which the wafers are mounted on the susceptor made of quartz, the heater chamber 405, a lower part of the wafers, and the vacuum chamber 406, an upper part, into which film growth gases
10 are to be introduced are separated.

[0040]

The inner walls of the vacuum chamber 406 are preferable to be set at a temperature by the foregoing heater 416 (wall heating means) not lower than a
15 temperature at which organometal gases can have sufficiently high vapor pressures and not higher than organometal gas decomposition temperature. That is, since organometal gases include several kinds of raw materials, the composition ratio of organometal gases
20 reacting on the wafer could be unbalanced owing to that a part or all of the organometal gases are liquefied or solidified or that organometal gases are decomposed, so that the temperature is preferable to be controlled not lower than the temperature at which the organometal gases
25 have sufficiently high dissociation speed without being condensed in the inner wall and lower than the decomposition temperature of the organometal gases.

Consequently, since the organometal materials and their decomposed products hardly adhere to the walls, generation of particles formed by peeling of the adhering substances can be prevented.

5 [0041]

In the present invention, it is preferable to coat the inner walls of the vacuum chamber with aluminum oxide since the coating is effective to further suppress the number of particles generated. The thickness of the
10 aluminum oxide is required to be thick enough to eliminate pin holes and preferably, for example, 1 μm or thicker, more preferably 3 μm or thicker, and furthermore preferably 5 μm or thicker. Nevertheless, it is ineffective to be too thick to suppress particle
15 generation, so that 10 μm thickness is sufficient, however there is no problem to be thicker than that. It is necessary for aluminum oxide to be formed firmly on the surface as to avoid separation and it is especially preferable to form aluminum oxide by sintering.
20 Incidentally, the thickness of aluminum oxide which is formed by natural oxidation on the aluminum metal surface is insufficient.

[0042]

In the present invention, though the inner walls of
25 the vacuum chamber may be coated directly with aluminum oxide just as described above, a liner 420 on whose surface aluminum oxide is formed may be disposed on the

inner wall faces of the vacuum chamber just as
illustrated in Fig. 3. In the case of using such a liner,
replacement is made easy, so that the particle generation
can further be suppressed by replacing the liner when
5 adhering substances, which are particle generation
sources, adhere to the liner.

[0043]

In this case, the liner may be disposed in all of
the inner walls of a vacuum chamber besides a vacuum
10 exhaust port and introduction ports of pipes.
Alternatively, the liner may be disposed only in the
parts where adhering substances exist especially much and
the wall surfaces of parts where no liner is disposed may
be coated with aluminum oxide. Further, aluminum oxide
15 may be formed previously in the inner walls of a vacuum
chamber and further a liner may be disposed on the
surfaces coated with aluminum oxide. Incidentally, that
a liner is disposed in the inner walls of a vacuum
chamber means a liner is placed in close contact with the
20 inner walls of the vacuum chamber to a certain extent.
But it is not necessarily required to be attached
perfectly closely to the inner wall to cover the walls.

[0044]

The material for the liner may be, for example,
25 quartz and aluminum. Since the structure of Fig. 3 is
same as that of Fig. 2 except the liner, the description
is omitted.

[0045]

In Fig. 2, the vacuum chamber 406 and the heater chamber 405 are evacuated by separate turbo molecular pumps 407, 408 and with such a structure, the leakage of organometal gases and an oxidizing gas to the heater chamber can be suppressed. Especially, in the case of this growth method using an oxidizing gas, oxidation of the heating mechanism 409 installed in the heater chamber to heat wafers can be prevented and thus the life of the heating mechanism 409 can significantly be prolonged. Such a structure is also effective to prevent short circuit and to prevent unevenness of the substrate temperature caused by adhesion of raw material gases on the heating mechanism. The pressure of the heater chamber during the film formation was 1×10^{-6} Torr when the pressure of a vacuum chamber was 1×10^{-3} Torr and the pressure difference of about three figures was obtained.

[0046]

When the temperature of a heater for heating wafers is increased, the temperature of a susceptor made of quartz is also increased to cause adhesion of organometal gases thereon, however by coating the surface of quartz with a metal oxide (a high temperature part-coating dielectric material) of such as STO (strontium titanate: SrTiO_3), the parting of adhering substances of organometal gases can be suppressed and as a result, contamination with particles can be prevented.

[0047]

As the high temperature part-coating dielectric material to be used in this case, the materials as described above may be used and further preferable materials are those which can firmly adhere to the material of a susceptor and have similar crystal system to that of a metal oxide dielectric film to be grown and are possible to form coatings by sintering. For example, STO can firmly adhere to quartz by sintering. Since PZT, the material to be grown, is a ceramic having the same crystal system as STO, the PZT is very difficult to be parted and particle generation scarcely takes place.

[0048]

Though this embodiment is described as the case of employing quartz as the material of a susceptor, besides quartz, SiC, Si, BN, or the like may be used and a high temperature part-coating dielectric material such as STO, BTO, PTO, PZT, BST, or the like can be sintered to form a coating on the surface.

[0049]

In the case a silicon wafer is used as a substrate, using Si as the material of a susceptor is preferable since temperature is easily made even in the wafer. For example, as silicon oxide film is formed on the surface of a silicon plate by thermal oxidation or the like and further STO is sintered on the resultant surface to obtain a susceptor made of silicon coated with a high

temperature part-coating dielectric material.

[0050]

In the present invention, it is satisfactory to coat the surface of a susceptor with a high temperature
5 part-coating dielectric material as described above, and of course, it is similarly effective that the whole body of the susceptor is made of such a material.

[0051]

Two lines, a main exhaust line 410 and a sub
10 exhaust line 411, are connected to an exhaust port of a vacuum chamber and the main exhaust line is connected to a turbo pump 407 through a main gate valve 412. The sub exhaust line 411 is connected to the turbo pump 407 through a valve 413 and then a water cooling trap 414.
15 During the film formation, the main gate valve 412 is closed and the valve 413 is opened to discharge exhaust gases through the sub exhaust line 411. With such a constitution, solidification and liquefaction of organometal gases in the turbo pump 407 can be avoided
20 and the life of the turbo pump 407 can be prolonged. Additionally, the total pressure of raw material gases in the vacuum chamber can be changed by introducing a valve 415 capable of adjusting conductance between the sub exhaust line 411 and the water cooling trap 414 and
25 adjusting the conductance. At the time when no raw material gas is supplied, the exhaust speed can be heightened by opening also the main gate valve 412 to

effectively maintain high vacuum degree. Further, these valves are effective to sharply decrease the partial pressure of each organometal gases in the vacuum chamber. The main gate valve 412 and the valve 413 are buried in a vacuum chamber 406 and enabled to be evenly heated.

[0052]

In this apparatus, though a turbo molecular pump is employed for a pump for exhaust, a mechanical booster pump, a dry pump, a rotary pump, or the like may be usable and it is required to plan the exhaust capability of the pump and conductance of the valves and the water cooling trap as to decrease the inner pressure of the vacuum chamber to 1×10^{-2} Torr or lower.

[0053]

A schematic diagram of a part of a raw material supply system is illustrated in Fig. 4. An organometal gas is a solid or a liquid at a room temperature and stored in a cylinder 501. The cylinder 501 is equipped with a valve 502 installed above and enabled to be parted from a flange 503 between the valve 502 and the supply system to exchange raw materials. Each organometal gas is extremely easily oxidized, so that the organometal gas is preferable to be kept no contact with atmospheric air. If being exposed to atmospheric air, the organometal gas is oxidized, a metal oxide is formed, the pipes are clogged, and a mass flow controller 504 is clogged.

[0054]

Organometal gases include those in a liquid state and those in a solid state at a normal temperature and normal pressure and the liquid raw materials are poured in cylinders as they are. On the other hand, solid raw materials are preferable to be deposited on the surface of alumina with about 1 mm diameter. By using such raw materials, gases can be generated stably at the time of heating. The purity of the generated gases is high.

[0055]

A pipe from the cylinder 501 is connected to the mass flow controller 504 through a stop valve 505 and thereafter divided into two pipes equipped with stop valves 506, 507, respectively, and connected to a vacuum chamber 508 and to a pump 512 via a water cooling trap 509, respectively. Parts of the raw material supply system comprising these pipes, the mass flow controller 504, valves 502, 505, 506, 507, and the likes and which are to be brought into contact with organometal gases are provided with heating means 513 and as same as the inner walls of the foregoing vacuum chamber, they are kept at a temperature not lower than the temperature at which the organometal gases are liquefied or solidified and not higher than the decomposition temperature of each organometal gas.

[0056]

The cylinder 501 is also enabled to be heated by the heating means 513 and heated to the extent where the

vapor pressure of each organometal material housed in the cylinder is high enough to operate the mass flow controller.

[0057]

5 At the time of film formation, at first the valve 502, 505, 506 are opened to operate the mass flow controller 504 by the spontaneous pressure of an organometal gas in the cylinder 501 and a gas is evacuated by the pump 512 to stabilize the gas flow rate
10 in the mass flow controller 504 during the time. Next, the valve 506 is closed and the valve 507 is opened, so that the organometal gas whose flow rate is precisely controlled can be supplied to the vacuum chamber.

[0058]

15 It is preferable for a vapor phase growth apparatus of the present invention to be provided with one series of the supply system as illustrated in the Fig. 7 for each organometal gas. Further, an exclusively separate pipe is preferable to be installed for an oxidizing gas.

20 [0059]

 In this embodiment, each of organometal gases and an oxidizing gas is introduced into a vacuum chamber through independent raw material supply pipes 418 (Fig. 1) and then mixed to one another in the vacuum chamber.
25 That is, the apparatus is so composed as to keep separated of the organometal gases and the oxidizing gas from each other before they are introduced into the

vacuum chamber. The reason for that is, because if the organometal gases and the oxidizing gas are brought into contact with each other, the organometal gases are oxidized and metal oxides are generated to cause clogging
5 of the pipes and clogging of the mass flow controller and further because the metal oxides are sent to the vacuum chamber to result in generation of particles and inhibition of thin film formation with excellent crystallinity at low temperature.

10 [0060]

The partial pressure of each organometal gas in the vacuum chamber is determined by the supply amount of each gas, the amount consumed on the substrate and the susceptor, the exhaust amount, and the adhesion amount to
15 the walls of the vacuum chamber. If the temperature of the walls of the vacuum chamber is kept at a proper temperature to keep a sufficiently high vapor pressure of each organometal gas and to inhibit decomposition of each organometal gas, the decrease owing to the adhesion of
20 the raw material gases to the inner walls of the vacuum chamber can be neglectable. Further since the raw material gas amount consumed on the substrate and the susceptor is as low as about 1/1000 of the supply amount, the pressure of the vacuum chamber is determined by the
25 gas supply amount and the exhaust amount. A vacuum gauge 510 for monitoring the pressure is installed in the vacuum chamber. Incidentally, the foregoing parts which

are to be brought into contact with organometal gases are required to be parts having sufficiently high heat resistance to the temperature at which at least organometal gases can have sufficient vapor pressure.

5 [0061]

Next, a preferable embodiment of double gate valves 104 (Fig. 1) will be described further in detail with the reference to Fig. 5. As illustrated in the Fig., double gate valves are separately connected to a vacuum chamber and an exchange chamber through the vacuum chamber side flange 301 and the exchange chamber side flange 302, respectively. In the vacuum chamber side, in the close state, the vacuum chamber side introduction route 307 is closed by contacting the vacuum chamber side valve 303 and the vacuum chamber side seal face 305 to each other. In the seal part, no O ring is employed and the metal faces are allowed to be contacted to each other. In the case of setting in the open state, the vacuum chamber side valve 303 is moved, for example, in the vertical direction to the plane of the Fig. by the valve operation part 311 to release the vacuum chamber side introduction route 307.

[0062]

In the exchange chamber side, in the close state, the exchange chamber side introduction route 308 is closed by contacting the exchange chamber side valve 304 and the exchange chamber side seal face 306 to each other

through O rings 309 for seal. Meanwhile in the open state, the exchange chamber side introduction route 308 is opened by moving the exchange chamber side valve 304 by the valve operation part 311.

5 [0063]

Further, the inside of the double gate valves is evacuated through the exhaust port 310 and kept vacuum. When a substrate is put in and taken out between the vacuum chamber and the exchange chamber, both of the vacuum chamber side valve and the exchange chamber side valve are opened and during the film formation in the vacuum chamber, both of the vacuum chamber side valve and the exchange chamber side valve are closed. By employing such a double gate valve structure, the following advantageous points can be provided. At first, it is made possible to suppress leakage of atmospheric air to the vacuum chamber at the time when the exchange chamber is released to the atmospheric air for wafer replacement. Also, it is made easy to keep high vacuum degree since no raw material adheres to the valve seal face in the exchange chamber side.

[0064]

Further, in the vacuum chamber side, metals are brought into contact with each other in the close state and there is no part to be deteriorated by heating to a high temperature, so that the temperature of the vacuum chamber can be set freely. If O rings are employed, in

the case a common O ring material is used, they are deteriorated by heating to, for example, around 180°C and it occurs a problem that the deteriorated material possibly becomes a particle generation source. On the other hand, since the exchange side valve is remote from the vacuum chamber at a high temperature, even if O rings are used, the O rings are not deteriorated and do not generate particles and the vacuum degree can sufficiently be maintained by the O rings.

10 [0065]

Described above is an example of employing double gate valves of the exchange chamber side valve, which is a general gate valve, and the vacuum chamber side valve, which is a gate valve with no O ring. However, the vacuum chamber side valve is not restricted to such a valve as long as it can shield the exchange chamber side valve from raw material gases. That is, it may be sufficient to install a movable shielding plate capable of shielding the flow of the raw material gases from the vacuum chamber side as to avoid direct adhesion of the raw material gases to a gate valve and such a movable shielding plate is sufficiently usable for practical use even if it does not have a completely closable property. Further, in order to prevent adhesion of the raw material gases, the movable shielding plate is preferably set at a temperature not lower than a temperature at which the organometal gases can have sufficiently high vapor

pressure and not higher than a decomposition temperature of the organometal gases.

[0066]

[Examples]

5 Next, an embodiment of a film formation employing the apparatus for vapor phase growth of the present invention will be described.

[0067]

Example 1

10 Investigation of the effect of the coating of the surface of a susceptor with a high temperature part-coating dielectric material was performed using a vapor phase growth apparatus of the present invention.

[0068]

15 Fig. 6 shows particle generation in the case of film formation carried out at 400°C for 40 seconds substrate temperature in conditions at first of 0.2 SCCM flow rate of $\text{Pb}(\text{DPM})_2$, 0.05 SCCM flow rate of $\text{Zr}(\text{OtBu})_4$, 0.25 SCCM flow rate of $\text{Ti}(\text{OiPr})_4$, and 3.0 SCCM flow rate
20 of NO_2 for nuclei formation and then in conditions of 0.25 SCCM flow rate of $\text{Pb}(\text{DPM})_2$, 0.225 SCCM flow rate of $\text{Zr}(\text{OtBu})_4$, 0.2 SCCM flow rate of $\text{Ti}(\text{OiPr})_4$, and 3.0 SCCM flow rate of NO_2 for film formation for 600 seconds. The Fig. 35 shows the correlation of the number of wafers on
25 which films are continuously formed and the total number of particles on 6-inch wafers in comparison using a quartz susceptor and using a quartz susceptor on which a

100 μm STO was formed by sintering. Here, the inner walls of the vacuum chamber were coated with aluminum oxide and kept at 180°C.

[0069]

5 According to Fig. 6, in the case where the
susceptor surface was quartz, the number of particles was
sharply increased when the number of wafers exceeds 1,000
sheets. That was attributed to parting of PZT stuck to
the quartz when the number of wafers exceeds 1,000 sheets.
10 On the other hand, in the case of forming sintered STO on
quartz, the increase of particles was slight. That was
attributed to that STO and quartz were firmly stuck to
each other and STO and PZT were ceramics both having
similar crystal systems and consequently, parting hardly
15 took place.

[0070]

Example 2

Next, raw material adhesion to the inner walls of a
vacuum chamber was investigated using a vapor phase
20 growth apparatus of the present invention.

[0071]

Respective plate specimens of aluminum, quartz,
quartz coated with a 10 μm thick aluminum oxide thereon
by sintering, stainless steel, copper, and silver were
25 made ready and these specimens were put in a vacuum
chamber kept at 180°C. When the respective plate
specimens were heated to 180°C, raw material gases were

continuously supplied to the vacuum chamber for 120 minutes in conditions of 0.25 SCCM flow rate of $\text{Pb}(\text{DPM})_2$, 0.225 SCCM flow rate of $\text{Zr}(\text{OtBu})_4$, 0.2 SCCM flow rate of $\text{Ti}(\text{OiPr})_4$, and 3.0 SCCM flow rate of NO_2 .

5 [0072]

As a result, copper was oxidized by NO_2 and corroded to be blacken on the surface. Though other materials were not corroded, a much amount of a white powder supposed to be the deposited organometal materials
10 adhered to the surface of quartz, the stainless steel, and silver. Further, though not so much as that in the case of quartz or others, a white powder adhered in the same manner to the aluminum surface to make it clear that an aluminum oxide in the thickness formed by spontaneous
15 oxidation was insufficient. In contrast with these materials, in the case of quartz coated with aluminum oxide by sintering, powder adhesion was not at all observed.

[0073]

20 Example 3

Next, investigation was performed for the effect on suppression of particle generation in the case of practically coating the inner walls of a vacuum chamber with aluminum oxide using a vapor phase growth apparatus
25 of the present invention.

[0074]

Fig. 7 shows particle generation state in the case

of film formation carried out at 400°C substrate temperature in conditions at first of 0.2 SCCM flow rate of $\text{Pb}(\text{DPM})_2$, 0.05 SCCM flow rate of $\text{Zr}(\text{OtBu})_4$, 0.25 SCCM flow rate of $\text{Ti}(\text{OiPr})_4$, and 3.0 SCCM flow rate of NO_2 for 5 40 seconds for film formation and then in conditions of 0.25 SCCM flow rate of $\text{Pb}(\text{DPM})_2$, 0.225 SCCM flow rate of $\text{Zr}(\text{OtBu})_4$, 0.2 SCCM flow rate of $\text{Ti}(\text{OiPr})_4$, and 3.0 SCCM flow rate of NO_2 for film formation for 600 seconds. The Fig. 36 shows the correlation of the number of wafers on 10 which films are continuously formed and the total number of particles on 6-inch wafers in comparison of the case of using aluminum oxide for the inner walls of the vacuum chamber and the case of using a stainless steel.

[0075]

15 A susceptor employed for the case was a quartz susceptor on which STO was formed by sintering and the inner wall temperature of the vacuum chamber was kept at 180°C.

[0076]

20 According to Fig. 7, in the case of using aluminum oxide for the inner walls, the number of generated particles was less even when the number of wafers exceeds 1,000 sheets. That was attributed to that the amount of raw material gases adhering to the walls was smaller in 25 the case of using aluminum oxide and consequently, the number of generated particles was lessened.

[0077]

Such results were observed in same in the case the walls of the vacuum chamber were lined with liners of quartz or aluminum both coated with aluminum oxide on the surface.

5 [0078]

Example 4

Next, investigation was performed on the relationship between the particle formation and the temperature of the inner wall of vacuum chamber. The feed of gasses was the same as in Example 1, a quartz
10 susceptor on which STO was formed was employed, and the inner wall of vacuum chamber was covered by aluminum oxide.

[0079]

15 Fig. 8 shows that the number of particles generated on the wafer was markedly lessened when the temperature of the inner wall of vacuum chamber was kept at a temperature of 150°C or more. A temperature of 175°C or more, particularly 180°C or more is much preferable.

20 [0080]

[Effect of the Invention]

By an apparatus of the present invention, an excellent metal oxide dielectric film can be formed while suppressing dust adhesion to a wafer at the time of film
25 formation, so that capacitors with miniaturized surface area and free of defects are made possible to be fabricated using a ferroelectric material and a high-

dielectric-constant material. Consequently, a semiconductor device with remarkably heightened integration degree can be fabricated at high production yield.

5 [BRIEF DESCRIPTION OF THE DRAWINGS]

Fig. 1 is a diagrammatic illustration of an apparatus for vapor phase growth of a thin film to be employed for the present invention;

10 Fig. 2 is a diagrammatic illustration showing the cross-section of a vacuum chamber of an apparatus for vapor phase growth of a thin film to be employed for the present invention;

Fig. 3 is a diagrammatic illustration showing the cross-section of one example of a vacuum chamber of a vapor phase growth apparatus of the present invention;

Fig. 4 is a diagrammatic illustration showing the raw material supply system of an apparatus for vapor phase growth of a thin film to be employed for the present invention;

20 Fig. 5 is a diagrammatic illustration of a double gate valve to be employed for the present invention;

Fig. 6 shows a graph illustrating the correlation of the number of wafers on which films are continuously formed and the total number of particles in the case of PZT formation on 6-inch wafers using a quartz susceptor and a quartz susceptor on which a 100 μ m STO is formed by sintering; and

Fig. 7 shows a graph illustrating the correlation of the number of wafers on which films are continuously formed and the total number of particles in the case of PZT formation on 6-inch wafers in the case of using
5 aluminum oxide for the material of the inner walls of the vacuum chamber and in the case of using a stainless steel for the material of the inner walls.

Fig. 8 shows a graph illustrating the total number of particles on each 6-inch wafer in the case of
10 continuously conducting film formation on 1,000 wafers while changing the temperature of the inner walls of the vacuum chamber;

Fig. 9 shows a graph illustrating the correlation between the film thickness and the spontaneous
15 polarization $2P_r$ in the case of PZT film growth on Pt using a sol-gel method;

[Reference Numerals]

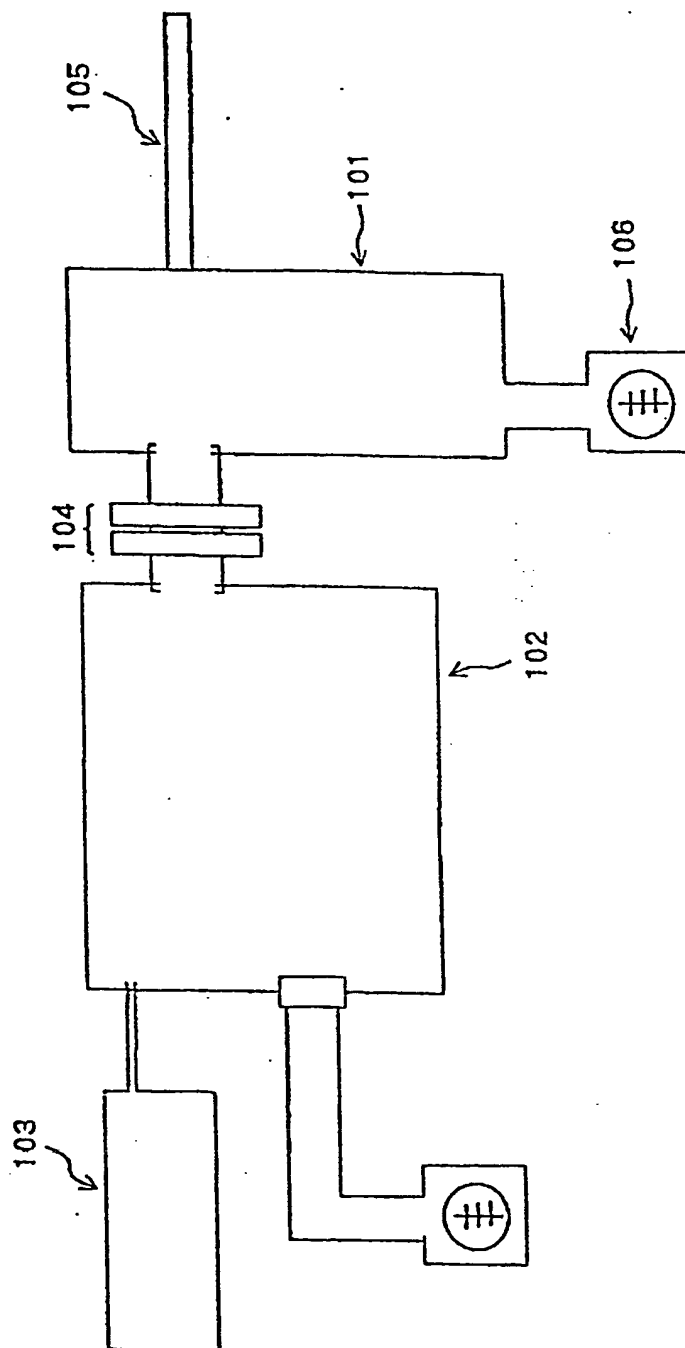
- 101 exchange chamber
- 102 vacuum chamber
- 20 103 raw material supply system
- 104 double gate valve
- 105 wafer transferring mechanism
- 106 turbo-pump
- 201 tehrmostat
- 25 202 bottle
- 203 organometal
- 204 carrier gas supply pipe

- 205 organometal material gas supply pipe
- 206 heating mechanism
- 207 growth chamber
- 208 wafer
- 5 209 gas exhaust
- 210 mass flow controller
- 301 vacuum chamber side flange
- 302 exchange chamber side flange
- 303 vacuum chamber side flange
- 10 304 exchange chamber side valve
- 305 vacuum chamber side seal face
- 306 exchange chamber side seal face
- 307 vacuum chamber side introduction route
- 308 exchange chamber side introduction route
- 15 309 O rings for seal
- 310 exhaust port
- 311 valve operation part
- 401 hole
- 402 quartz pin
- 20 403 susceptor
- 405 heater chamber
- 406 vacuum chamber
- 407 turbo molecular pump
- 408 turbo molecular pump
- 25 409 heating mechanism
- 410 main exhaust line
- 411 sub exhaust line

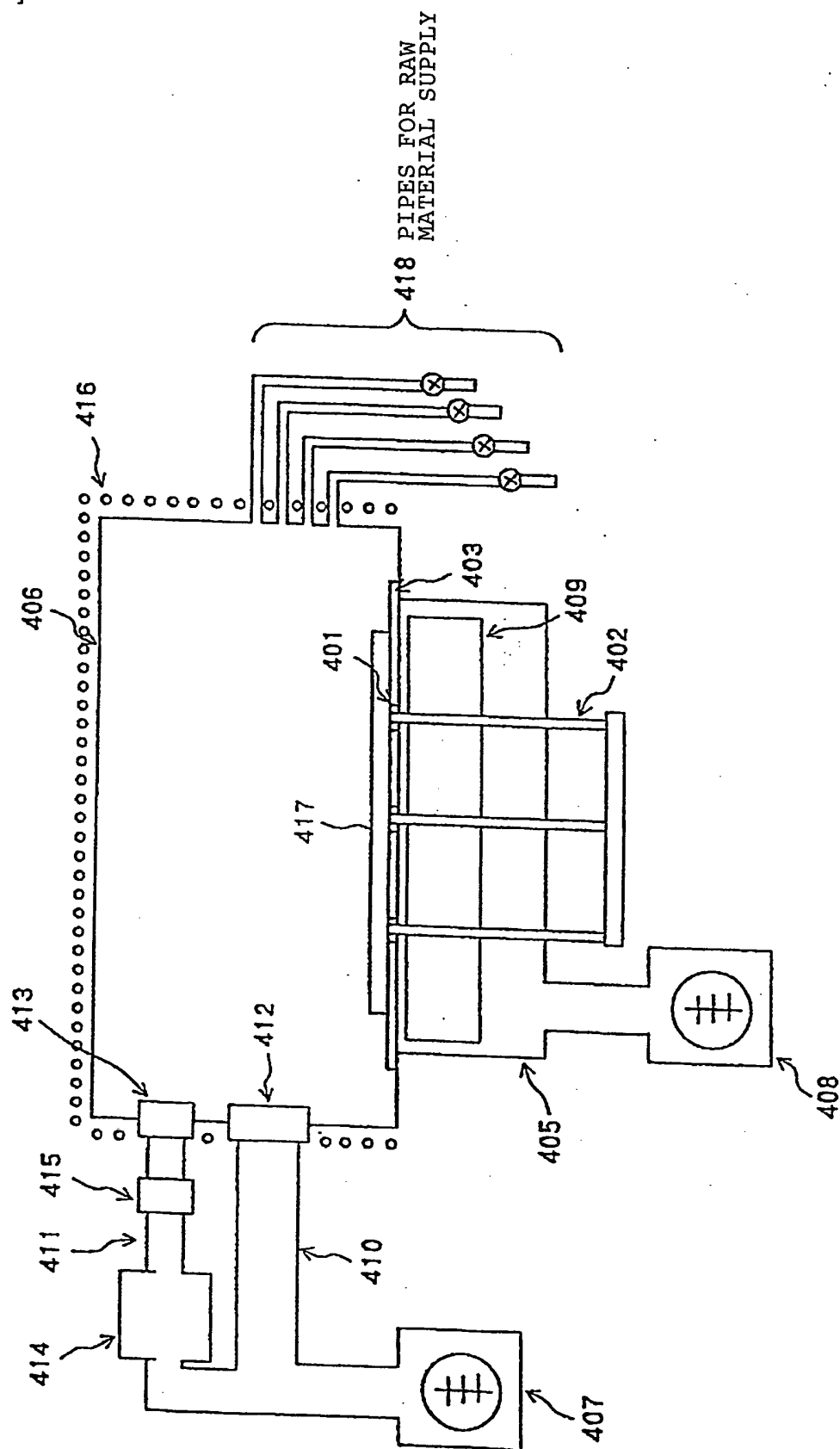
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413 valve
414 water cooling trap
415 conductance valve
5 416 heater
417 wafer
418 raw material supply pipe
420 liner
501 cylinder
10 502 valve
503 flange
504 mass flow controller
505 stop valve
506 stop valve
15 507 stop valve
508 vaccum chamber
509 water cooling trap
510 vacuum gauge
512 pump
20 513 heating means

[Document Name] Drawings

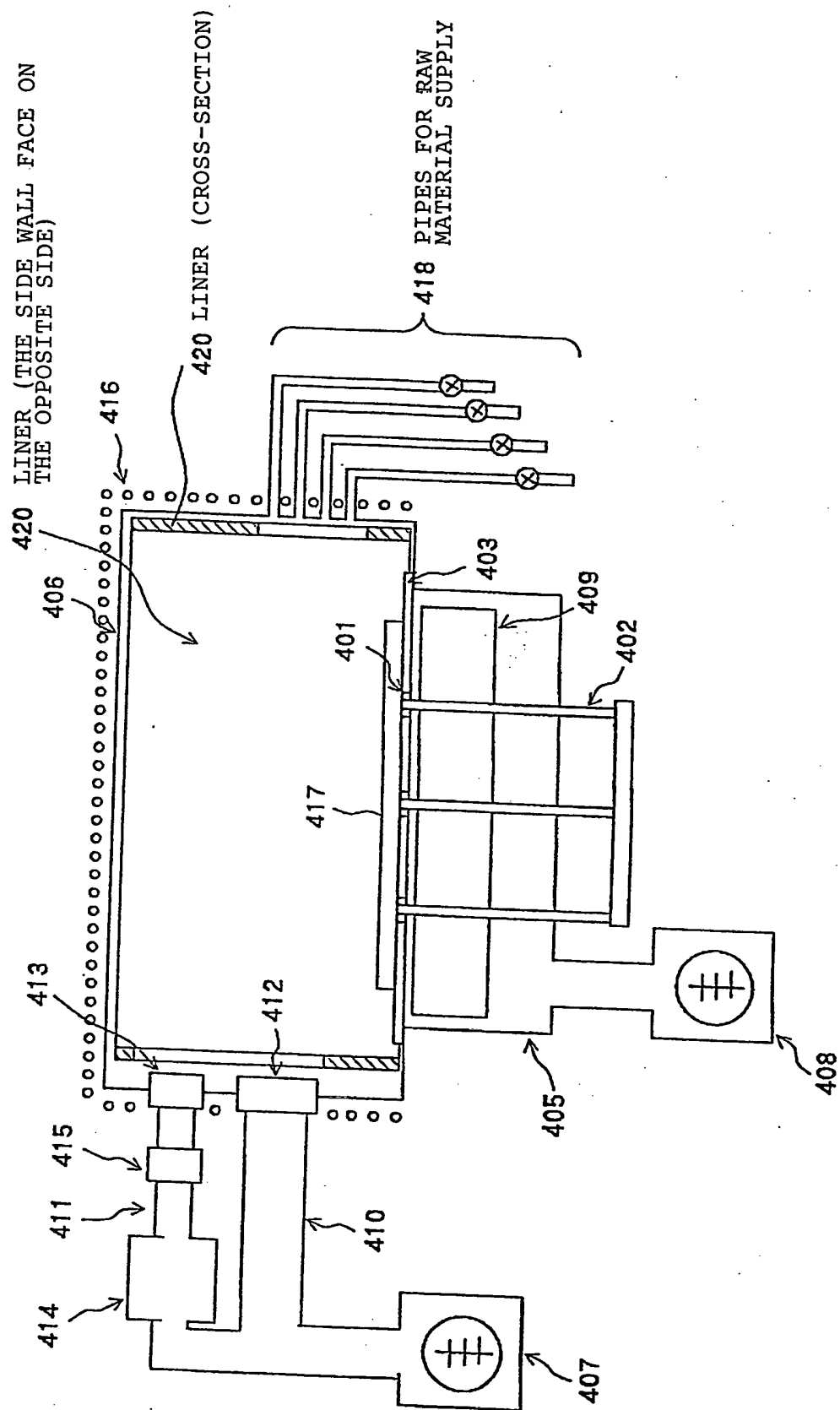
[Fig. 1]



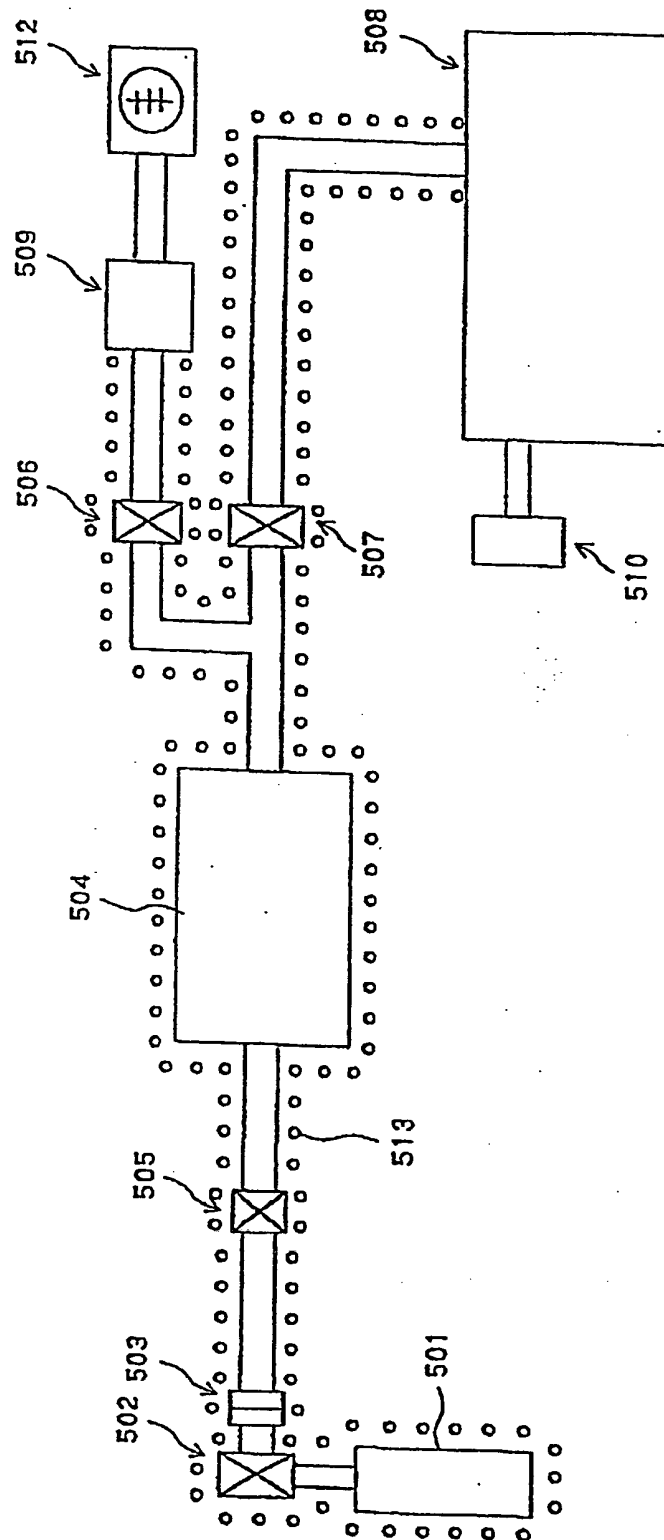
[Fig. 2]

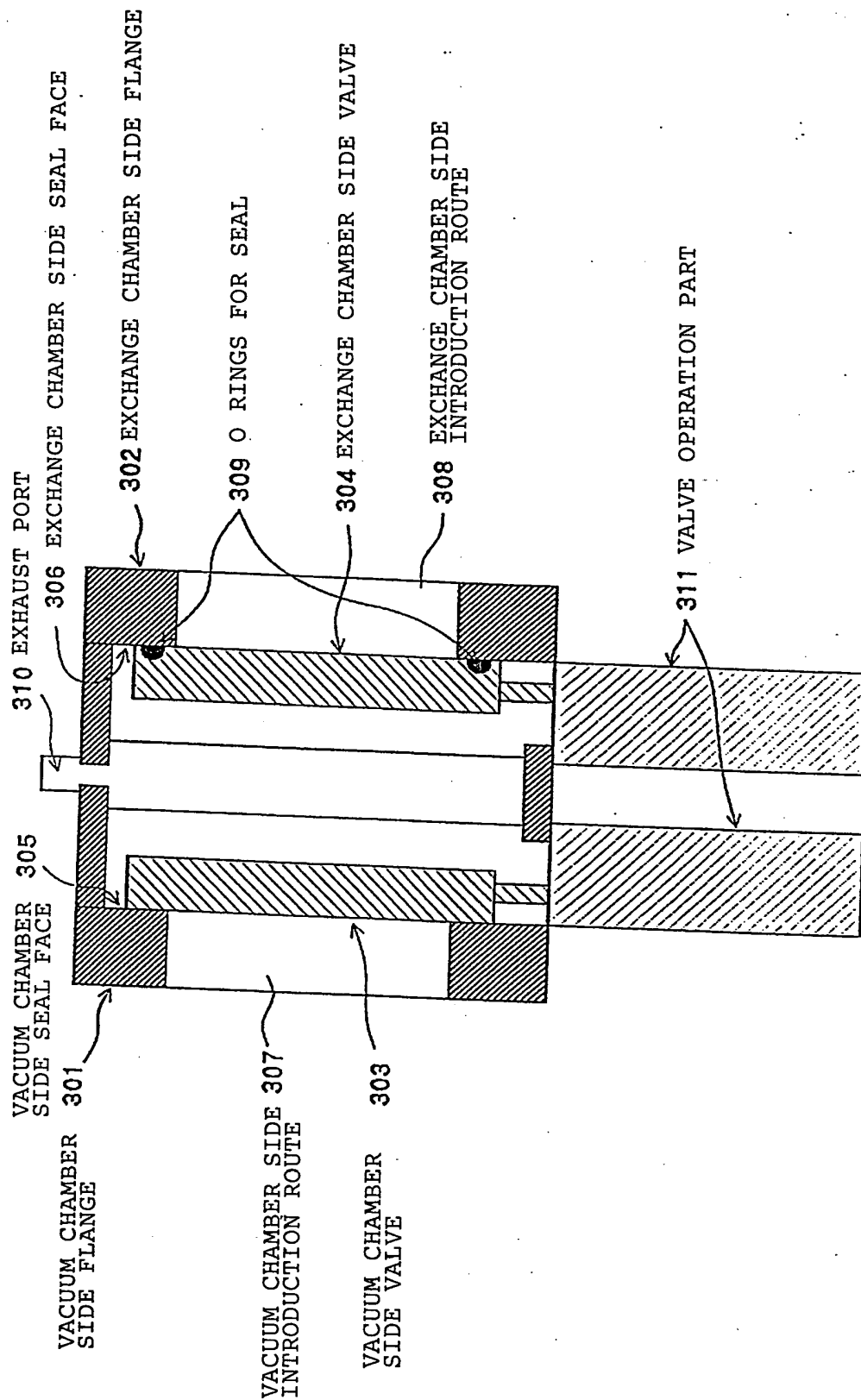


[Fig. 3]

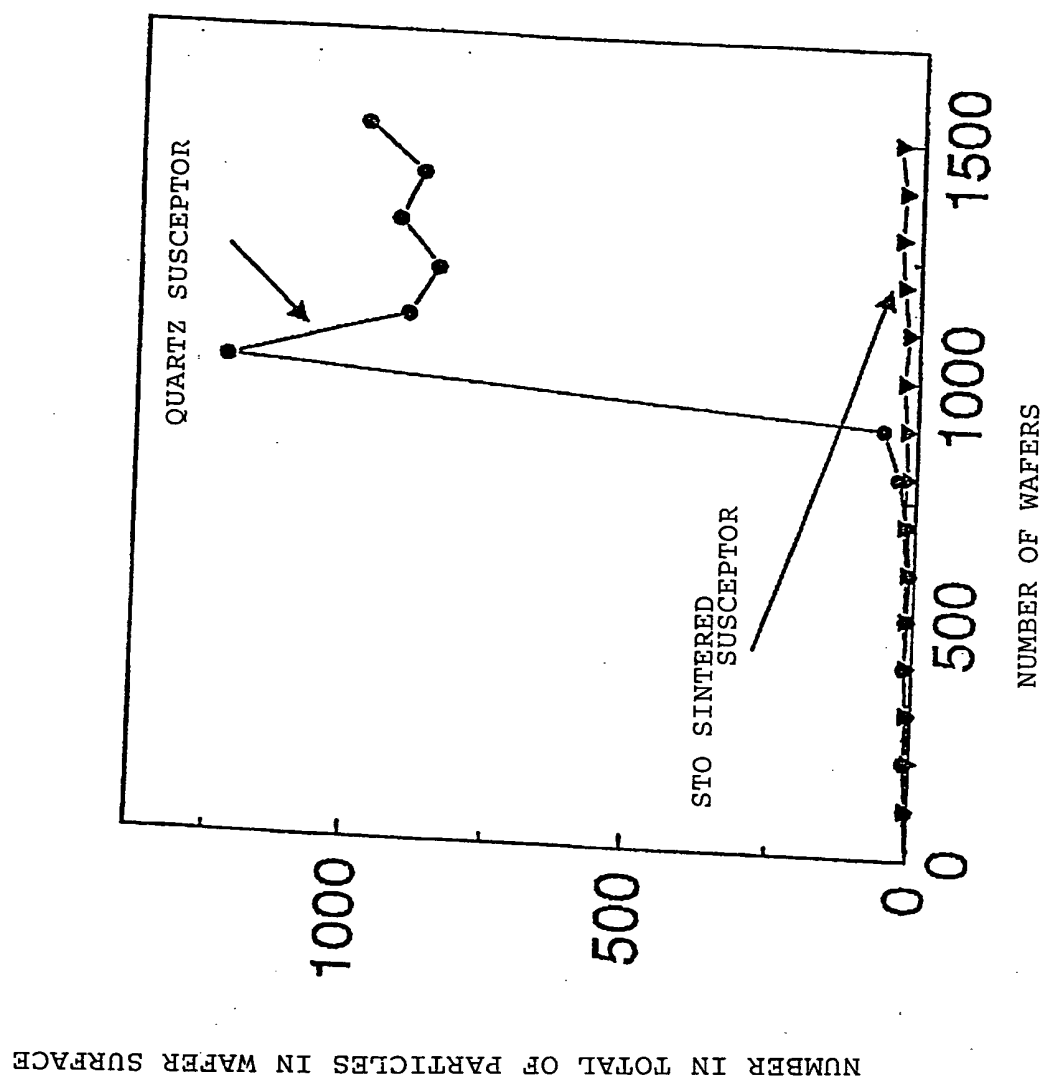


[Fig. 4]

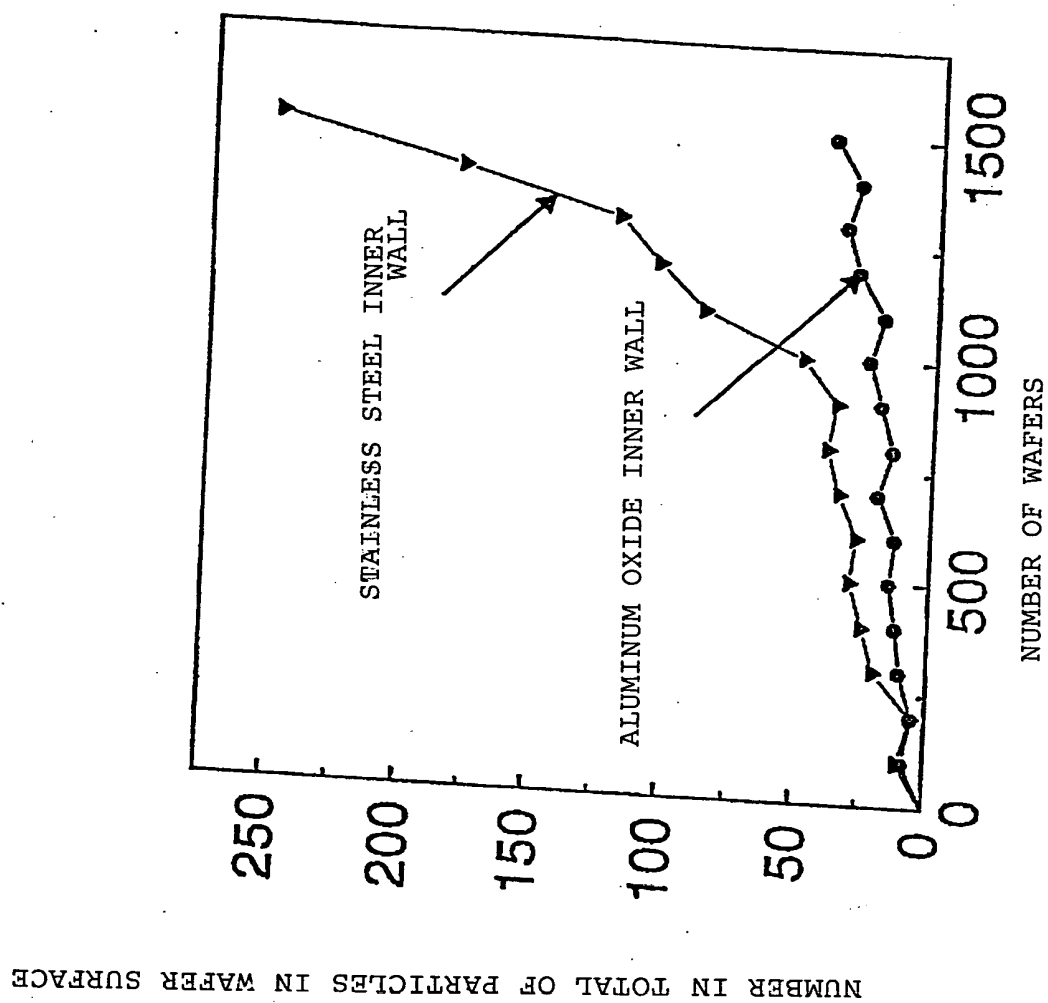




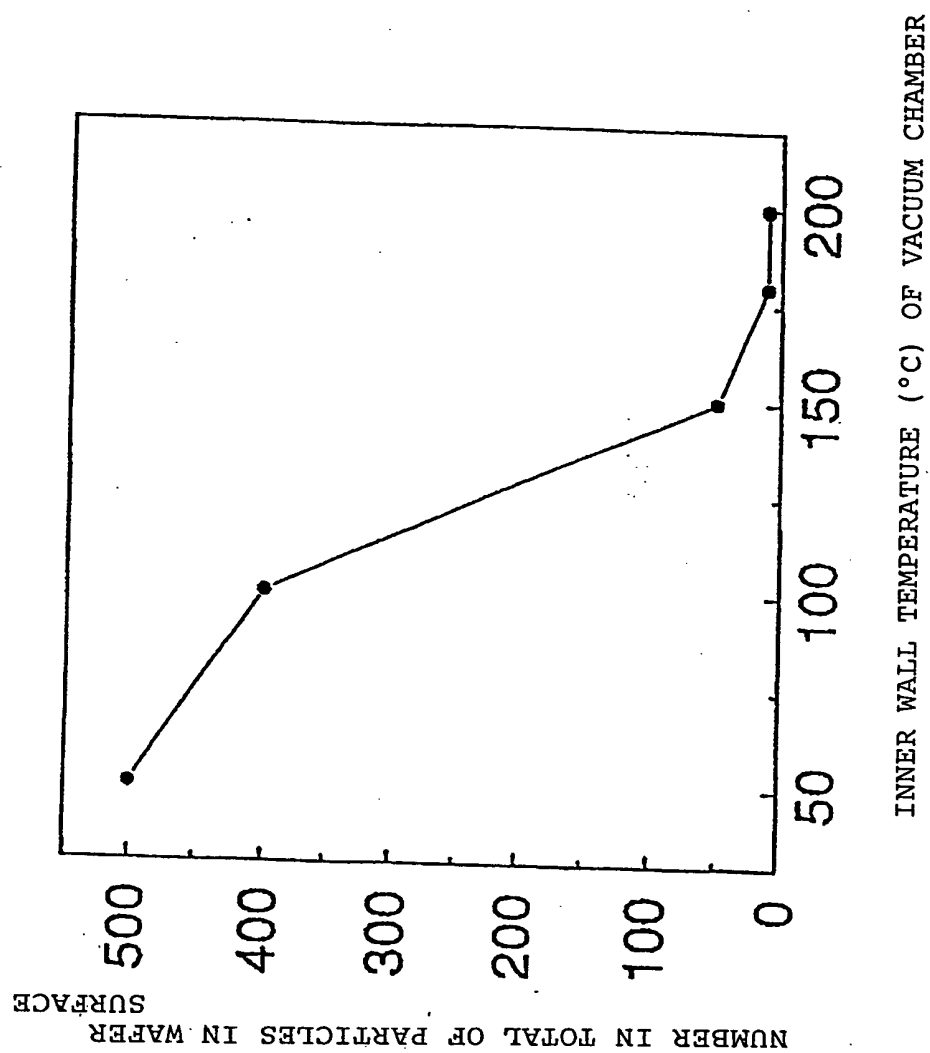
[Fig. 6]



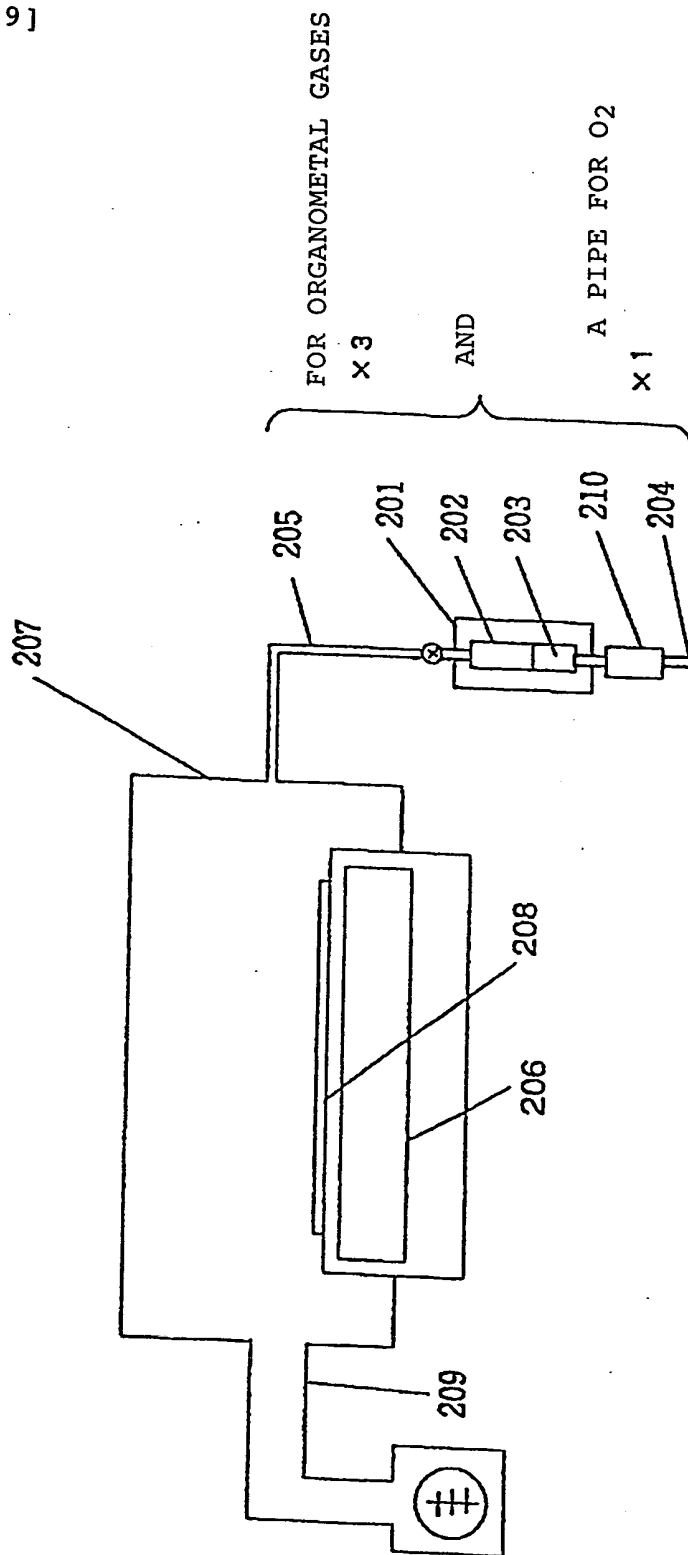
[Fig. 7]



[Fig. 8]



[Fig. 9]



[Document Name] Abstract

[Abstract]

[Problem] The present invention aims at providing a
vapor phase growth apparatus capable of forming a metal
5 oxide dielectric film with little dust adsorption by
suppressing dust generation during the film formation.

[Solution] An apparatus for use in vapor phase growth
of a metal oxide dielectric material on a capacitor
electrode using organometal gasses, wherein such parts in
10 vacuum chamber 406 that contact with the starting
material gasses and have a temperature of the
decomposition of starting material gasses or higher - at
least the surface in contact with the starting gasses of
susceptor 403 on which wafer 417 mounts - are composed of
15 a metal oxide dielectric material which may be the same
as or different from the objects of vapor phase growth.

[Selected Drawing] Fig. 2